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EXPERIMENTAL INVESTIGATION OF FAILURE OF HIGH STRENGTH STEEL ON ACCOUNT OF EXTERNAL HYDROGEN EMBRITTLEMENT & EFFECT ON MECHANICAL PROPERTIES

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ABSTRACT

High strength steel is used in a various engineering& industrial applications. Industries like Automobile, Aerospace, Power & Energy, Oil, Marine, Defence etc. using high strength steel to develop new lightweight energy efficient solutions with improved efficiency and lower overall weight of structure. High strength steels are subjected to a perennial problem of hydrogen embrittlement during course of manufacturing and under specific condition of operation resulting in unexpected failure of components below permissible stress limit and without any prior warning severely amputating the service life. This failure is predominantly brittle in nature & result in catastrophic accident. Seriousness of issue demands a systematic analysis of parameters responsible for hydrogen embrittlement by simulating the failure in a controlled environment using laboratory tests. EN24 steel, which is an alloy steel having nickel, chromium & molybdenum as main alloying elements offers good machinability and claims a good high strength with good resilience, hardness and wear resistance. EN24 steel is primarily used in automotive, machine tool and power generating equipment industries for manufacturing of frames, structural members, fasteners, power transmission shafts, axles, landing gears etc. This paper details the investigation taken up to simulate the failure of EN24 steel specimen subjected to external hydrogen embrittlement by cathodic pre-charging of specimen in acidic environment and testing the specimen using conventional as well as controlled strain rate test technique. The objectives of the experiments are to understand the influence of various external parameters, to establish the simples test/ assessment methodology and to generate the baseline data for further establishing correlation between operational performance and laboratory testing resulting reduction in life leading to unexpected failure.

KEYWORDS: High Strength Steel, External Hydrogen Embrittlement & Brittle failure

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Original Article

1. INTRODUCTION

1.1 Hydrogen Embrittlement

Originally introduced in year 1875 by Johnson [1], the presence of atomic hydrogen leads to the reduction in ductility of a material is known as hydrogen embrittlement [2]. The hydrogen embrittlement phenomenon as per ASTM F2078 is defined as “An irreversible loss of ductility in a metallic alloys caused due to infusion of hydrogen with combining stresses, whether its internally or externally applied residual stresses”. Hydrogen embrittlement (HE) may result to transform high strength (HS) steel from ductile to brittle by absorbing and subsequent distribution of hydrogen in microstructure making them susceptible to fracture. Hydrogen available in atomic form may easily enter into atomic structure and damage high strength steel. Hydrogen can enter into high strength steel

during galvanizing, pickling, cleaning, electroplating, phosphating and in the service environment [3]. When atoms of hydrogen get infuse into the alloys such as aluminium, steel, titanium alloys and some other alloys, this causes changes and reduced load carrying ability and reduced ductility.

The fracture phenomena uses the term hydrogen embrittlement caused due to the infusion of hydrogen atoms & also acknowledged in the form of hydrogen infused failure or hydrogen atomic blistering. Those material which are highlysensitive coversHS steels, Ti and Al alloys and & Cu [4]. Embrittlement due to hydrogen might deteriorate mechanical properties and leads to delayed breakdown which can take place due to externally applied stresses or internal residual stresses and at times ductile material becomes brittle [5]. Additionally, the mechanical properties may further deteriorate due to slow straining which indicates time affectedinfusion as a contributing parameter. HE is usually the take place at ambient temperature. Though at elevated temperatures, hydrogen may get extractedout of microstructure.

Various parameters influencing the mechanical conduct of steel are presence of hydrogen, exposure condition to hydrogen environment, temperature, surface conditions, chemical composition & microstructure of steel, strain rate etc[6].

HS steel is usually sensitive to hydrogen attack, therefore HE is caused due to this phenomena [9-34]. High strength steel fasteners used in industries are classified on basis of tensile strengths which range of between 1,000–1,500 MPa, and these steels are frequently used for critical industrial components and failure of these can have serious results [3]. Some prior conditions for hydrogen embrittlement to occur is the interaction of load carrying HS steel to a significant volume of hydrogen, that results in losing mechanical properties such as toughness and ductility along with propagating brittle crack. [8, 9]. Hydrogen embrittlement may sometimes cause reduce in ductility without major decrease in tensile and yield strength [10] but certainly degradation in elongation occurs.

Additionally, HE sensitivity of HS steel increases with increasing strength, reduction in loading rate, presence of large inclusions etc[11-14]. Hydrogen interaction with metals under stress is highly complex and various mechanisms are proposed by different authors has been recapped by Lynch [15,16,17].

Factors which are responsible for Hydrogen embrittlement are [4, 18] :-

- Tensile & yield Strength of material.
- Microstructure of a material.
- Amount of residual stresses
- Temperature, Pressure & exposure time to hydrogen environment.
- Stress & Strain rate during service & application.
- volume of hydrogen.
- Molecular defects such as voids, trap, grain boundary defects etc.
- Presence of coatings on metal surface.
- Susceptibility to acidic solutions.
- Heat treatment during manufacturing.

In this paper HE susceptibility of high strength steel (EN24) has been studied in an simulated environment with hydrogen pre-charging using cathodic reaction at various potentials.

1.2. Mechanism of Hydrogen Embrittlement and Characteristics

A critical amount (ppm) of atomic hydrogen is required for embrittlement of HS steel and their mechanical properties are susceptible under H environment. Metals those are subjected to embrittlement are HS alloyed steels, Manganese steel, alloyed Al, Mg, Ti and its alloys etc [19-24]. Hydrogen embrittlement is complex process for which various mechanisms and failure theories are proposed. There are variety of mechanisms available developed by various researchers, some are (i) hydrogen enhanced de-cohesion mechanism (HEDE), (ii) Hydrogen enhanced local plasticity model (HELP), (iii) adsorption-induced dislocation emission (AIDE), (iv) Hydrogen changed micro-fracture mode (HAM) (v) Hydrogen Enhanced Macroscopic Ductility (HEMP) (vi) hydride formation, [16,22,25-28]. Different blend of these mechanisms also take place for deterioration and embrittlement of metals. In spite of various known theories, real process that initiate embrittlement in metal is an ambiguity but researchers are further putting efforts to recognize primary causes of a hydrogen embrittlement.

1.2.1. Hydrogen Enhanced De-cohesion (HEDE)

Troiano in 1959 proposed that this is the most simplified mechanism. This mechanism suggest that a critical amount of hydrogen diminishes the cohesive strength or interatomic bond strength between de-cohesion and atoms in the region of the crack opening occur when the critical crack tip opening is reached [15,16,17,26,29,30]. With the existence of external stresses hydrogen atoms are diffused into the molecular structure and results in reduce in inter-atomic strength or cohesive strength at the tip of the crack and forms cleavage like fracture. The difficulty in proving this model is the measurement of cohesive forces [31].

1.2.2. Hydrogen Enhanced Localized Plasticity (HELP)

This work was carried out in 1972 which is the most practical model to represents the mechanisms of hydrogen embrittlement, which consider the interaction of hydrogen and plasticity. Model suggested that the hydrogen accumulated near the tip of the crack reduce the dislocation resistance, resulted in high mobility of dislocation and operate as an accelerator for plastic warping[31,32]. With the presence of hydrogen, the dislocation movement is likely to occur at low value of load due to confined reduction in yield stress. However, exact form of fracture may depend on the amount of hydrogen, stress intensity of crack tip and microstructure. Fractographic tests also indicates a localized plastic deformation with reduced macroscopic ductility[17,18,33-36,39]. This mechanism is appropriate for the material with FCC, BCC structures, alloys material etc [17].

1.2.3. Adsorption Induced Dislocation Emission (AIDE)

Presented by Lynch in 1976, Aide mechanism propose that the H atoms accumulated near surface in the high stress region like crack tips, which reduces the inter-atomic bond and cohesive strength by HEDE and enable the dislocation formation from crack root and further propagation by slip and creating the voids by HELP [16, 31]. In this mechanism crack formation and movement occurred due to anti-cohesion and dislocation at the crack root. Due to mixed effect of slip at the crack root the crack propagation and fracture occurs with voids amalgamation [16,17,33,38,39].

1.2.4. Hydrogen Enhanced Macroscopic Ductility (HEMP)

As per this model of HE, Hydrogen available in large concentration influence the mechanical characteristics of steel due to solid solution tempering & hydrogen diffusion by hydrogen atoms. Failure and yielding plastic deformation take place over

complete specimen volumetric length results in macroscopic enrichment of plasticity. In this mechanism, yield strength reduction occurs because of the occurrence of large volume hydrogen is termed as Hydrogen Enhanced Macroscopic Ductility [40, 41, 42].

1.2.5. Mixed Fracture (MF)

Fracture surface examination shows that the fracto-graphy includes both ductile (MVC) and brittle(fisheye) features. Therefore, it is said that the ultimate fracture of material has been resulted by the mixed effect of these failure mechanisms. The fish eye propagation take place radially until they match with micro-void coalescence. These fish eyes defects are surrounded by micro void cracks. Explained HE failure mechanism termed as mixed fracture mechanism.

1.2.6. Hydrogen Assist Micro Void Coalescence (HDMC)

It is a ductile fracture mechanism, which propose that the crack propagation occur in stages such as void formation &growth, void coalescence, void nucleation, crack enhancement and failure of remaining section by shear fracture. This mechanism states that the crack propagation take place in a blunt pattern by joining the defect voids generated due to hydrogen diffusion. Micro void coalescence generates dimples with reduced ductility. Ultimate fracture appears on account of shear stresses in low shear dimple present along with some brittle inter-granular fracture at the boundaries of the specimen [39,44,45].

1.3 Characteristic of Hydrogen Embrittlement

With the studies carried out in previous researches it was suggested that the following conditions must are responsible for the reduction in mechanical properties caused by HE –

- Materials with high tensile strength steel 10.9 grade steel or (UTS>1000MPa).
- Existence of Hydrogen in alloys or in surrounding.
- Development of severe tensile stresses during operational condition.
- The value of hardness on surface should be higher than HRC 37.

2. MATERIAL & METHODOLOGY

Commercially available ready to use hardened and tempered high strength steel EN24 / AISI 4340 is used to analyse the effect of HEunder different condition of hydrogen charging as well as zinc plating. EN24T steel having good machinability and claims a good high tensile steel strength with resilience, hardness and wear resistance. It has a tensile strength of 800/1200 MPa. EN24 being used in the automobiles and tool industries for fasteners, gears, shafts, pinions, spindles etc.

Chemical and mechanical properties of EN24 as determined at independent laboratory are indicated in Table 1 and 2. The material used with two heat treatment conditions namely T & X differentiated by heat cycle and UTS. The specimens for tensile and impact test are prepared as per ASTM E8 and E23.

Table 1: EN24Chemical Properties

Carbon	0.35-0.45%	**Silicon**	0.10-0.35%
Nickel	1.30-1.80%	**Manganese**	0.45-0.70%
Chromium	0.90-1.40%	**Phosphorus**	0.05% max
Molybdenum	0.20-0.35%	**Sulphur**	0.05% max

Table 2: EN24Mechanical Properties

Grade	U.T.S. (MPa)	Yield (MPa)	Elongation (%)	Impact(KCV) J	HardnessBrinell
T	**850-1000**	**650**	**13**	**35**	**248-302**
U	925-1075	855	12	42	269-331
V	1000-1150	750	12	42	293-352
W	1075-1225	940	11	35	311-375
X	**1150-1300**	**1020**	**10**	**28**	**341-401**
Y	1225-1375	1095	10	21	363-429
Z	1550	1235	5	9	444

Grade T and X are used for preparation of test specimen and experiments

Tensile, impact and fatigue tests are performed with specimens in following conditions –

- Normalized (U.T.S- 700 MPa, Y.S. – 550 MPa)
- Zinc plated (U.T.S- 700 MPa, Y.S. – 550 MPa)
- Hardened& Tempered (U.T.S - 1300 MPa, Y.S. – 1020 MPa)
- Zinc plated Hardened& Tempered (U.T.S - 1300 MPa, Y.S. – 1020 MPa)
- Hydrogen Pre-charged Hardened& Tempered (U.T.S - 1300 MPa, Y.S. – 1020 MPa) with six different combination of current and charging time.

A set of six specimens are used to perform each of tensile, impact and fatigue test to investigate the behaviour under different conditions amounting to 150 nos. total specimens.

Table 3: Testing Conditions for Tensile, Impact & Fatigue Test

Condition	Material Grade	Description	Pre H-Charging Parameters
1	EN24 Normalized (U.T.S- 700 MPa, Y.S. – 550 MPa)	Normal	No H-Charging
2	EN24 Normalized (U.T.S- 700 MPa, Y.S. – 550 MPa)	Zinc Coated	No H-Charging
3	EN24 Normalized (U.T.S- 700 MPa, Y.S. – 550 MPa)	H-Charged	1A/24 hr.
4	EN24 Normalized (U.T.S- 700 MPa, Y.S. – 550 MPa)	H-Charged	1A/48 hr.
5	EN24 Normalized (U.T.S- 700 MPa, Y.S. – 550 MPa)	H-Charged	1A /64 hr.
6	EN24 Normalized (U.T.S- 700 MPa, Y.S. – 550 MPa)	H-Charged	1.5A /24 hr.
7	EN24 Normalized (U.T.S- 700 MPa, Y.S. – 550 MPa)	H-Charged	1.5A /48 hr.
8	EN24 Normalized (U.T.S- 700 MPa, Y.S. – 550 MPa)	H-Charged	1.5A /64 hr.
9	EN24 (Hardened and Tempered) (U.T.S - 1300 MPa, Y.S. – 1020) MPa)	Normal	No H-Charging
10	EN24 (Hardened and Tempered) (U.T.S - 1300 MPa, Y.S. – 1020) MPa)	Zinc Coated	No H-Charging
11	EN24 (Hardened and Tempered) (U.T.S - 1300 MPa, Y.S. – 1020) MPa)	H-Charged	1A/24 hr.
12	EN24 (Hardened and Tempered) (U.T.S - 1300 MPa, Y.S. – 1020) MPa)	H-Charged	1 A/48 hr.

Table 3: Contd.,

13	EN24 (Hardened and Tempered) (U.T.S - 1300 MPa, Y.S. – 1020) MPa)	H-Charged	1 A/64 hr.
14	EN24 (Hardened and Tempered) (U.T.S - 1300 MPa, Y.S. – 1020) MPa)	H-Charged	1.5/24 hr.
15	EN24 (Hardened and Tempered) (U.T.S - 1300 MPa, Y.S. – 1020) MPa)	H-Charged	1.5/48 hr.
16	EN24 (Hardened and Tempered) (U.T.S - 1300 MPa, Y.S. – 1020) MPa)	H-Charged	1.5/64 hr.

For assessment of HE performance of the material, tensile tests of charged specimen are carried out at controlled strain rate in air. Hydrogen charging of the specimen has been done by cathodic reaction. 0.5 M H2So4 Solution was prepared. During preparation of solution, a concentrated H2So4 Solution is mixed with water is right proportion. In a 1 litre of volumetric flask, 53.3 ml of concentrated H2So4 Solution is required. By using this a dilute H2So4 Solution is prepared and cathodic reaction performed for hydrogen diffusion in high strength steel. For cathodic reaction, test specimen is made cathode and platinum wire is used as anode. Hydrogen changing done with current density of 100 A m^{-2}. The tensile test conducted30mins after hydrogen-charging process was finished.

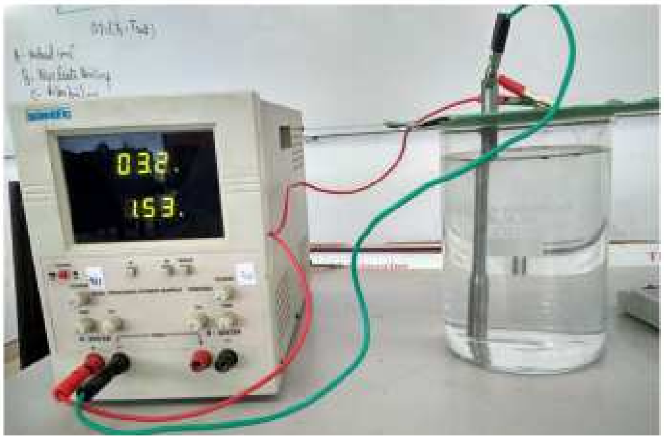

Figure 1: Hydrogen Charging.

Figure 2: Rotating Bending Fatigue Test

Tensile, Impact and Fatigue tests are conducted with above test specimens and results are summarised below.

Further fatigue test (rotating bending) & Impact (Charpy) are performed on pre-charged test specimen with charging parameters as indicated in table 3.

3. RESULTS & DISCUSSIONS

Results of tensile, Impact and Fatigue tests are summarised below in graphical form-

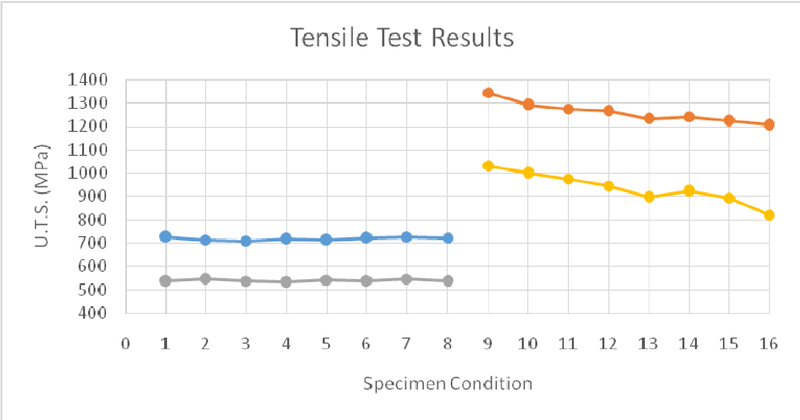

Figure 3: Tensile & Yield Strength Comparison for Various Condition of EN 24 Material.

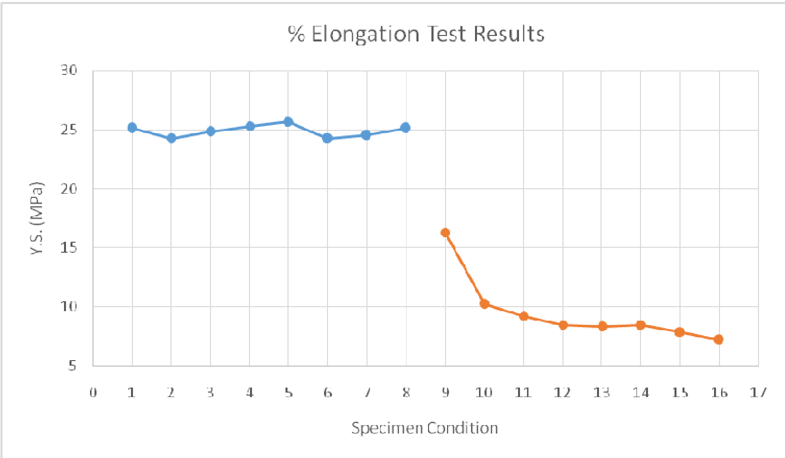

Figure 4: Elongation Comparison for various Condition of EN 24 Material.

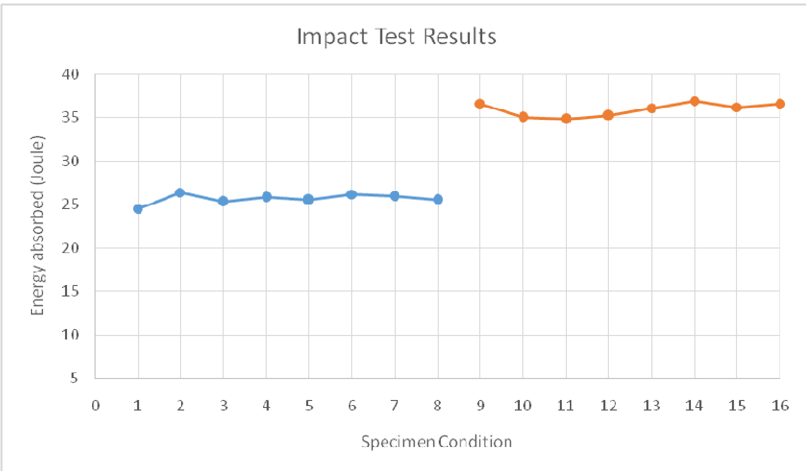

Figure 5: Impact Strength Comparison for Various Condition of EN 24 Material.

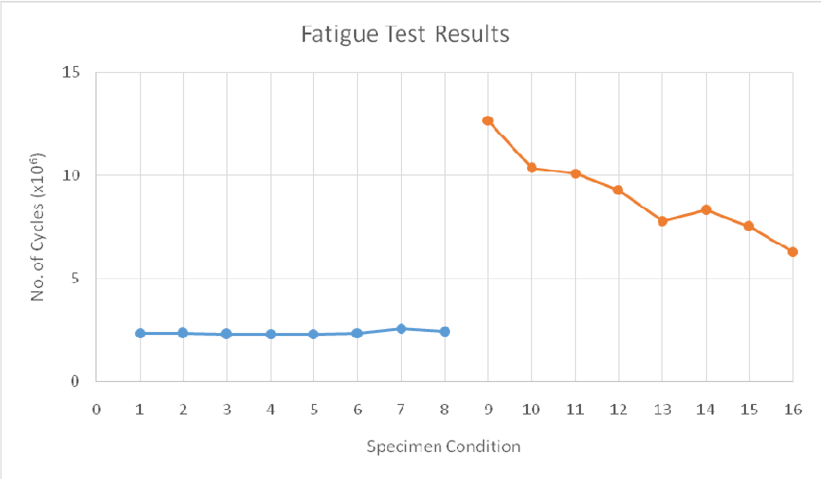

Figure 6: Fatigue Life Comparison for various Condition of EN 24 Material.

4. DISCUSSION ON RESULTS OF EXPERIMENTS

4.1 Tensile Test

- For Normailzed EN24 (HRC22) specimen the tensile and y.s. of bare and zinc plated specimen are not changed by significant amount.
- Whereas, hardened &tempered EN24 specimens with no Zinc plating shows different and dissimilar rersults for Yield, UTS and percentage elongation when comparing the mechanical properties (Yield, UTS and % elongation) with Zinc coated/ plated.
- The plated specimen has less values of Yield and UTS than the unplated specimen, which indicate that due to zinc plating, weakening of specimen takes place.
- Further a declining trend is observed for tensile and yield strength with increased H-charging current and time indicating higher hydrogen diffusion rate.

4.2 Elongation

Elongation percentage for the zinc coated specimen (HRC 42) is approximately 8%, whereas its value for unplated specimen recorded around 16%. Reported reduction in percentage elongation due to zinc plating indicate that the brittleness occurs in zinc plated specimen due to hydrogen diffusion in lattice at the time of picking process before plating was done.

- There is significant reduction in % elengation for Hardened and Tapmered specimen after Zn Coating.
- Results indicate that the zinc plating has significantly reduced the % elongation indicating reduction in ductility.
- H-charging has a prominent detrimental effect upon the ductility in terms of reduction in % elongation.
- A drastic reduction in % elongation is observed with the increase in charging current and charging time.
- Reduction in % elongation may be attributed to higher hydrogen diffusion in microstructure.

4.3 Impact Strength

- As documented in literature review it is found that hydrogen presence in the microstructure has no effect on impact strength.
- This may be attributed to fact that sudden loading does not allow the hydrogen atoms to affect the fracture mechanism.
- As there is no significant change in impact properties, this can be concluded the hydrogen embrittlement does not affect material properties for impact loading condition.
- From the results, it was found that the energy variation during all experimental work has not changed widely and variation was found to be ±4%. So it is concluded that the impact strength of material has not affected widely due to hydrogen diffusion.

4.4 Fatigue Life

- The fatigue life results indicate a downward trend in the results for HS steel EN24 specimen.
- With the increasing charging time and charging current fatigue life found to be decreased significantly.
- Fatigue crack progress found predominantly high in the H-charged samples with respect to crack progress rates in without charged specimens.
- Hydrogen introduced in microstructure of EN24 steel may be attributed to have reduced the fatigue life.

5. FRACTURE CHARACTERISTICS

Type of fractures seen during failure of test specimen are: (i) ductile cup-cone failure and (ii) shear fracture as shown in Figure 7a & 7b. Necking observed in cup & cone fracture of mild steel charged and high tensile steel charged specimen indicates ductile failure.

Figure 7(a&b): Fracture Modes After Tensile Tests.

Figure 3b indicates Brittle Fracture with very little necking having three zones as: (i) crack initiation; (ii) Crack Propagation; and the (iii) Final Fracture Region.

6. MICROSTRUCTURE ANALYSIS

Microstructural analysis gives a significant indication of HE susceptibility. Scanning electron microscopy (SEM), optical microscopy and transmission electron microscopy (TEM) are the most widely used microstructural analysis technique for characterization of material and for fractographic examination of fractured specimen. SEM is generally

preferred as it is simple in use and versatile with easier sample preparation process as compared to others techniques [46]. For identifying defects such as dimples, microvoids and fisheyes SEM examination is mostly used and preferred technique [47].

Figure 8, displays the effect of hydrogen changing vide surface morphology of H-charge EN24 steel specimen. The image shows presence of severe hydrogen bubbles on the surfaces of high strength steel alloy EN24. The severity of hydrogen blisters depends on the time of charging and amount of current during H-charging]. The extent of hydrogen attack is linked to the increasing of the cathodic charge time & current density.

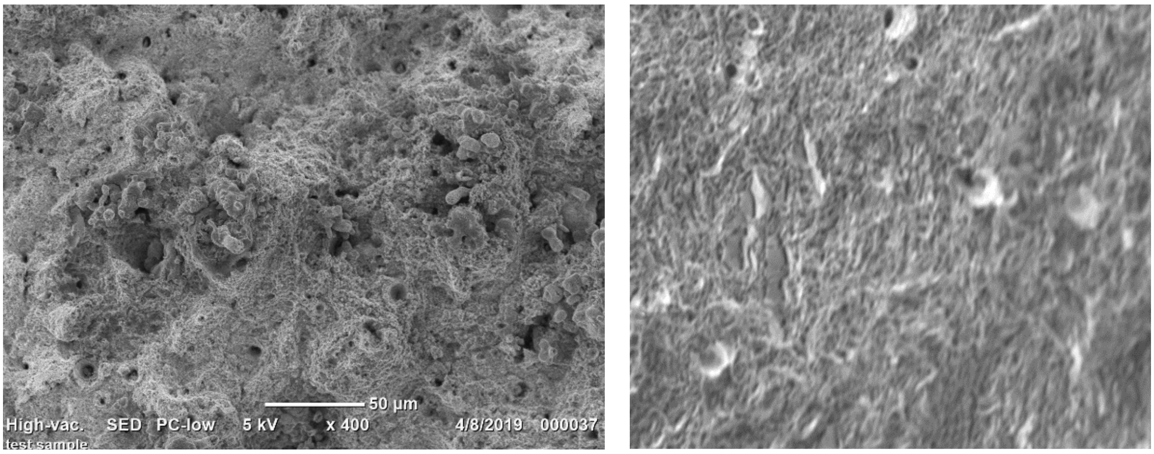

Figure 8(a&b): SEM images of Fractured Surfaces After Tensile Tests.

An SEM image (Figure 8b) of the failed surface after tensile testing of the uncharged sample shows a fractured surface which is clean and without much micro-voids. On the other hand, failed surfaces found in the H exposed specimen (Figure 4a) are characterized by dimples &quasi-cleavage fractured surface. It is reported in literature that such failure seen in hydrogen exposed fracture found having dimples with irregular fracture and defects. Here complete fractured surface consists of failure features closely observed with brittle failure.

Figure 9 shows fracture images (SEM) of EN24 steel after fatigue testing for both hydrogen-charged and uncharged specimens. In the charged sample (Figure 9b) the fracture surface appears with some irregular lumps showing a quasi-cleavage fracture. In the hydrogen-free specimen (Figure 9a) the fracture surface appears more clean and ductile.

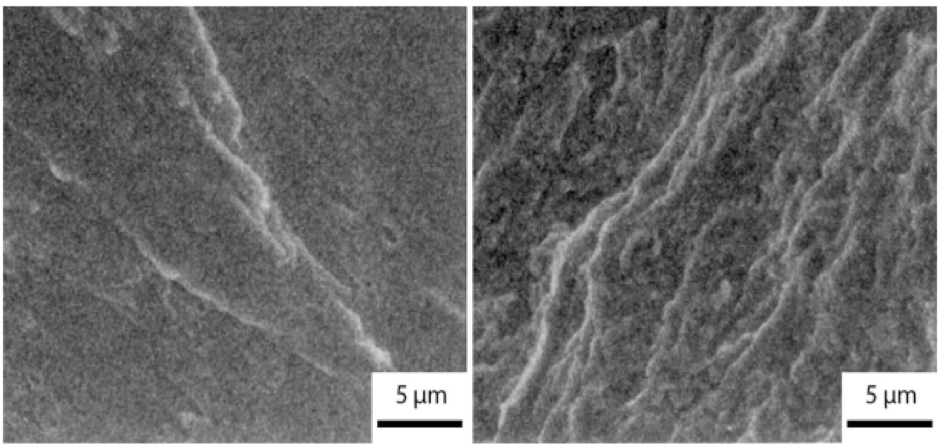

Figure 9(a&b): SEM Images of EN24 Steel After Fatigue Test.

7. SUMMARY& CONCLUSIONS

The experiments conducted investigates the behaviour of H-induced failure ofEN24 martensitic steel in tensile, impact and fatigue loading condition. The key findings are summarised as follows:

- Tensile tests revealed that the H-charged samples are less ductile and failed with lesser elongationin comparison to uncharged samples.
- Failed surface of the H-charged samples shows shallow dimple patterns and quasi-cleavage patterns. Most of the fractured surface covered with voids, which implies the presence of hydrogen affected defects formation.
- The declining trends in Tensile and yield strength with charging time and currents shows that increased hydrogen diffusion resulting in faster crack growth.
- Considerable change in % elongation indicates transformation from ductile to brittle fracture regime and the same is established with fracture morphology.
- Impact strength is not affected by h-charging as this does not allow the diffused hydrogen to affect the fracture pattern owing to very high strain rate.
- H-charging of EN24 steel having a predominant effect on fatigue life. Fatigue crack growth rates in the hydrogen-charged were accelerated with respect to crack growth rates in uncharged specimens.

Experiments performed on EN24 steel specimen in various condition of H-charging provide an insight of hydrogen embrittlement related failure and fracture mechanism.

Further efforts shall be made to prevent the high strength materials (HSS) from HE when working in hydrogen environment or atmosphere. Present study explains the effect of hydrogen in mechanical properties (Yield, UTS and % elongation), impact strength and fatigue life of EN24 material. Mechanism responsible for this degradation in properties were discussed. Future work shall focus on finding the suitable coatings to minimize the HE of high strength steel for industrial applications.

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